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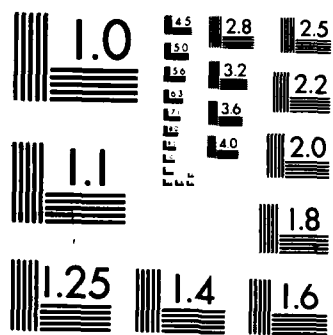
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OFFICE OF NAVAL RESEARCH

Contract N00014-83-K-0470-P00003

Task No. NR 359-718

TECHNICAL REPORT # 47

In Situ Infrared Spectroelectrochemistry

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Prepared for Publication in

Journal of Vacuum Science and Technology B

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July 30, 1986

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1. REPORT NUMBER 47	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) In Situ Infrared Spectroelectrochemistry		5. TYPE OF REPORT & PERIOD COVERED Technical Report # 47
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Stanley Pons, C. Korzeniewski		8. CONTRACT OR GRANT NUMBER(s) N00014-83-K-0470-P0003
9. PERFORMING ORGANIZATION NAME AND ADDRESS University of Utah Department of Chemistry Salt Lake City, UT 84112		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Task No. NR 359-718
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Chemistry Program - Chemistry Code 472 Arlington, Virginia 22217		12. REPORT DATE July 30, 1986
		13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) IR spectroelectrochemistry, infrared radiation		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The vibrational spectrum of molecules at or near an electrode surface can be obtained using subtractively normalized interfacial Fourier transform infrared spectroscopy (SNIFTIRS) and electromodulated infrared spectroscopy (EMIRS).		

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J. Vac. Sci. & Tech., B

3 (1985) 1421

### *In situ* infrared spectroelectrochemistry

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The vibrational spectrum of molecules at or near an electrode surface can be obtained using subtractively normalized interfacial Fourier transform infrared spectroscopy (SNIFTIRS) and electromodulated infrared spectroscopy (EMIRS). The large electric field existing at the electrode/solution interface is sufficient to induce dipole moments in highly polarizable molecules. The magnitude of the induced dipole moment is proportional to the electric field strength. As a result of this interaction some totally symmetric normal vibrations are made infrared active, and other bands appear in violation of the surface selection rule. This effect is known as the electrochemical Stark effect. Calculation of the expected absorption coefficients for the totally symmetric modes of adsorbed species gives values which are close to those observed experimentally. Experimental evidence of  $A_g$  mode activation of adsorbed pyrene is presented. The electric field dependence of the integrated absorption coefficient is demonstrated for acrylonitrile adsorbed on a gold electrode.

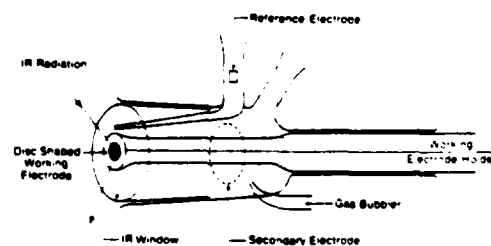
In recent years several techniques have been developed for obtaining *in situ* the infrared spectra of species at the electrode-solution interface.<sup>1,2</sup> The vibrational spectra is strongly dependent upon the molecular orientation with respect to the electrode surface. Electromagnetic radiation polarized perpendicular to the metal surface (*s* polarized) undergoes a phase shift close to 180° for all angles of incidence resulting in a standing wave which has little amplitude at the metal surface. The electric vectors of light polarized parallel to the plane of incidence (*p* polarized) add constructively upon reflection at glancing angles giving rise to a sizable component of electric vector normal to the surface. Therefore, only molecules which have a component of their dipole moment normal to the surface are able to interact with electromagnetic radiation.

Since intense electric fields exist near electrode surfaces, it is possible for the electric field to interact with highly polarizable molecules. This interaction may distort the diffuse electron cloud inducing a dipole in the direction of the electric field. If the polarization of the infrared radiation is in the same direction as the induced dipole, the integrated absorption coefficient for the transition is proportional to the square of the electric field strength. Therefore, the electric field at the electrode surface may allow infrared activity for vibrations normally forbidden by symmetry or the surface selection rule. This effect is known as the electrochemical Stark effect.<sup>6</sup> Perturbation of vibrational spectra by strong electric fields was first predicted by Condon<sup>7</sup> and has been demonstrated in several systems.<sup>8-10</sup>

10-mm-<sup>diameter</sup> diam platinum or gold disk mounted on the end of a glass syringe barrel. The electrodes were polished to a mirror finish using alumina of decreasing sizes down to 0.05  $\mu\text{m}$ , then positioned in the cell so as to trap a thin layer of solution between the metal surface and the single crystal silicon infrared transmission window. The cell was then mounted in the path of a focused infrared light beam such that the angle of incidence from the normal was slightly less than 70 °C.

Spectra were obtained using both subtractively normalized Fourier transform infrared spectroscopy (SNIFTIRS) and electromodulated infrared spectroscopy (EMIRS).<sup>12</sup>

In the standard SNIPTIRS technique, interferograms are collected and coadded at each of two electrode potentials. After transformation of the interferograms to the frequency domain spectra, the two results are ratioed. The final spectrum represents the difference in the infrared vibrational structure of the interfacial region at the two potentials. If transmission notation is chosen for the display of the spectrum, then the result is in units of  $1 + \Delta R/R$ , where  $\Delta R/R$ , the normalized reflectivity, is composed of the difference in reflectivity of the surface region at the two potentials, and  $R$  is the reflectance of the electrode at the potential corresponding to the state used in the denominator of the original



## II. EXPERIMENTAL

Experiments were performed in a three-electrode thin-layer cell (Fig. 1). The working electrode consisted of an 8-

FIG. 1 Design of the thin layer spectroelectrochemical cell used for EMIRS and SNIFTIRS experiments.

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ratio. At small values of  $\Delta R$  compared to  $R$ ,  $\Delta R/R$  corresponds to the absorbance  $A$ . <sup>+ through pot</sup>

In EMIRS, a high-throughput dispersive monochromator is used. The potential at the electrode is rapidly modulated between the potentials of interest, thereby modulating the relative amounts of absorbing species in the interfacial region. This results in a small modulation of the reflectivity which is easily extracted by synchronous demodulation techniques. Details of both experimental techniques have been presented earlier.<sup>1,7</sup>

For cleaning cells, triply distilled water was used for steaming previously acid-cleaned cells. Aqueous solutions were prepared from recrystallized salts and triply distilled water. Acetonitrile (Burdick and Jackson, Muskegon MI) was used as received. The water content was nominally <0.01%. Organic substrates were obtained from Aldrich and used as received. Electrochemistry was controlled by a potentiostat and waveform generator (JAS Instrument Systems, Inc.). Cells and electrodes were also obtained from JAS.

Typically, spectra were obtained at an electrode potential where the species of interest was either not adsorbed or weakly adsorbed; and at a potential where the species was present on the electrode surface or interfacial region to a large extent. The values of these potentials can be obtained usually by conventional double layer capacitance measurements which can give quantitative values of the surface coverage and information on the thickness of the electrical double layer near the interface.

### III. RESULTS AND DISCUSSION

#### A. Calculation of $B$

In an experiment where the electric field ( $\vec{E}_z$ ) is applied in a direction  $z$ , the induced dipole moment becomes

$$\mu_z = \alpha_{zz} \vec{E}_z,$$

where  $\alpha_{zz}$  is the polarizability tensor. The integrated absorption coefficient is given by

$$B = \frac{2\pi^2 \nu T}{\epsilon_0 h c} \mu_A^2,$$

where  $\mu_A$  is the transition dipole matrix element,  $T$  is the number of absorbing molecules per unit area in the beam path, and all other constants have their usual meaning. In the presence of an external electric field the transition dipole matrix element can be expressed as the sum of the permanent

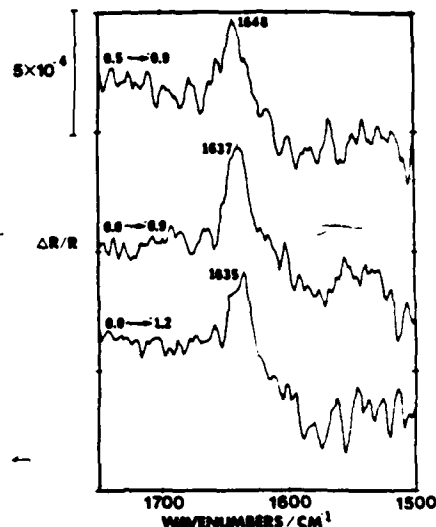


FIG. 2. SNIFTIRS difference spectrum of 0.5 mM pyrene at a platinum electrode in acetonitrile with 0.1 M tetra-n-butylammonium tetrafluoroborate (TBAF) as supporting electrolyte as a function of potential vs  $Ag^+/Ag$ .

dipole moment ( $\mu_0$ ) and the induced dipole moment ( $\mu_z$ ) where

$$\mu_0 = \sum_i \langle r_i \rangle.$$

Thus,

$$|\mu_A| = \langle \Psi_f | \mu_0 + \mu_z | \Psi_i \rangle.$$

If the expression for  $\mu_0$  and  $\mu_z$  are expanded in a Taylor series and the high order terms are neglected the integrated absorption coefficient becomes:

$$B = \frac{2\pi^2 \nu T}{\epsilon_0 h c} \langle \Psi_f | \mu_0' \cdot \Psi_i \rangle + E \langle \Psi_f | \alpha' \cdot \Psi_i \rangle$$

where  $\mu_0'$  and  $\alpha'$  correspond to the change in the permanent dipole moment and polarizability with respect to a normal coordinate, respectively. We have calculated the absorption coefficient for the  $C = C$  symmetric stretch of molecules adsorbed flat at an electrode surface. For these molecules  $\mu_0$  approaches zero and we are left with evaluating the remaining matrix element. Since the matrix elements  $\langle \Psi_f | \alpha' \cdot \Psi_i \rangle$  are not tabulated for molecules larger than diatomic, we have estimated this change to be of the same order of magnitude as the polarizability normal to the molecular axis, providing an upper bound value to the matrix element. The polarizabilities necessary for the calculation were taken from Ref. 11. The value of the absorption coefficient is calculated as a function of electric field strength and shown in Table I.

#### $A_g$ mode activation

We have observed electric field activation of the totally symmetric  $A_g$  mode of pyrene at or near the surface of a platinum electrode. Figure 2 is the SNIFTIRS spectra of 0.5

TABLE I. Predicted values for B.

$E$ V/m	$C_6H_6$	Naphthalene	Anthracene	TCNQ <sup>-</sup>	Pyrene
$10^6$		0.056	0.1	1.5	0.4
$10^5$	0.6	5.6	11	150	36
$2 \times 10^4$	2.4	22	44	580	140
$5 \times 10^3$	15	140	280	3600	900
$8 \times 10^2$	40	350	710	9300	2300
$10^2$	62	560	1100	14500	3600

TABLE I. Predicted values for B.

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potential. In systems where perpendicular adsorption is certain, such as CO adsorbed on platinum and  $\text{CN}^-$  adsorbed on silver, band position is a linear function of potential.

Although the fit is only approximate it is sufficiently good to demonstrate the effect of strong electric fields on infrared spectra. Intense electric fields present near the electrode surface may interact with polarizable molecules and induce a dipole moment in the direction of the field which give rise to very weak absorptions. With refinements in the calculation it may be possible to use the electrochemical Stark effect to study the electric field intensity in the double layer region of the electrode.

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